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TARGET MADE OF HIGH-PURITY METALLIC TANTALUM AND PROCESS FOR
ITS PRODUCTION

Inventors:

Iwao Kyono [JP/JP]
Sogokenkyusho
Nippon Mining Co., Ltd.
3-17-35 Shinsonan
Toda, Saitama 335 (JP)

Hiroshi Hosaka [JP/JP]
c/o Sogokenkyusho
Nippon Mining Co., Ltd.
3-17-35 Shinsonan
Toda, Saitama 335 (JP)

Seiji Yaegashi [JP/JP]
c/o Sogokenkyusho
Nippon Mining Co., Ltd.
3-17-35 Shinsonan
Toda, Saitama 335 (JP)

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Translated from Japanese by the Ralph McElroy Translation Company
910 West Avenue, Austin, Texas 78701 USA

Applicant:

Nippon Mining Co., Ltd. [JP/JP]
1-12-32 Akasaka
Minatoku, Tokyo (JP)

Agents:

Motohiro Kurauchi et al.,
patent attorneys

Designated States:

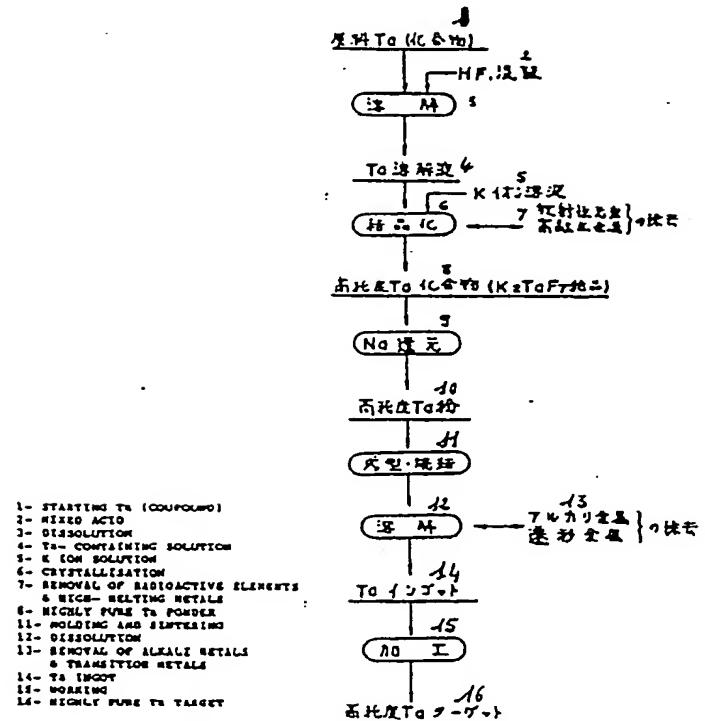
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International search report

Abstract

A target made of highly pure metallic tantalum having only extremely reduced amounts of alkali metals, radioactive elements, transition metals, and high-melting metals harmful for semiconductor devices. The target contains up to 50 ppb (0.05 ppm) of alkali metals, up to 5 ppb (0.005 ppm) of radioactive elements, up to 3 ppm of transition metals, and up to 3 ppm of high-melting metals. A process for producing the target is also disclosed. It comprises a combination of a wet purifying step mainly involving precipitation of potassium fluorotantalate (K_2TaF_7) crystals and sodium reduction and a subsequent drying step. Sputtering using this target enables production of a high-quality Ta_2O_5 insulating film and a metallic Ta electrode film.



Key: 9 Na reduction
10 High-purity Ta powder

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Technical field

The present invention concerns a target made of a pure metallic tantalum (TA) and a process for its production, more specifically concerns a high-purity metallic tantalum target for sputtering used in the formation of a tantalum oxide film (Ta_2O_5 film) for semiconductor devices and a process for its production. The Ta_2O_5 films formed using the tantalum target of the present invention have an extremely low content of impurities that are harmful to semiconductor devices and are highly suitable for insulation films in semiconductor devices. Also, the tantalum target of the present invention is suitable for LSI electrode formation.

Technical background

Conventionally, silicon oxide films (SiO_2 films) have been used as insulation films between electrode wirings in semiconductor devices. However, due to the requirements for thinner insulation films for a high degree of integration in LSI, the performance of silicon oxide films have become unsatisfactory, and the use of tantalum oxide film (Ta_2O_5 film) with a high dielectric constant has been tried. Currently, great attention has been focused on Ta_2O_5 with a high dielectric constant as the dielectric material for capacitors in high-capacity MOS dRAMs (dynamic random access memory). Such Ta_2O_5 films are usually formed by sputtering a tantalum target in an argon-oxygen mixed gas. However, the sputtered Ta_2O_5 thin film contains a large number of trap centers, and leakage current may flow readily. The main cause of such leakage current is thought to be due to residual impurities. Thus, to reduce leakage current, reducing the content of residual impurities is essential.

Also, to improve the reliability of performance of semiconductor devices, it is necessary to reduce the content of impurities such as (1) alkali metals such as Na, K, Li, etc.; (2) radioactive elements such as U, Th, etc.; and (3) transition metals such as Fe, Cr, Ni, Mn, etc. Alkali metals such as Na, etc. migrate easily in the gate insulation films, which will result in deterioration of the interface properties. The radiation from radioactive elements such as U, etc. has a fatal effect on the operation reliability of semiconductor devices. This, called soft errors, is a very important matter in the computer industries. Transition metals such as Fe, etc. impart important effects, generating an interfacial level and cause contact leakage.

Due to such reasons, reducing the content of impurities such as alkali metals, radioactive elements and transition metals is essential for use as constitutional materials for VLSI.

In addition to the above Ta_2O_5 films, metallic tantalum films are being investigated for use as electrode films such as LSI gate electrodes, source electrodes, and drain electrodes. Conventionally, polycrystalline silicon is used for such electrodes, but problems of signal transport delay and MOS element formation by self-aligning occur, and using high-melting metals having a lower resistance than polycrystalline silicon and silicides thereof has been

widely tried. Tungsten, molybdenum, etc. as well as tantalum are promising candidates. Needless to say, even for such tantalum electrodes, reducing the content of impurities is essential.

Films from either metallic tantalum or tantalum oxide are formed mainly by the sputtering method and vapor deposition method. In the sputtering method, argon ions collide with a metallic target plate to release the metal, and the released metal is deposited on a substrate facing against the target plate. On the other hand, in the electron beam vapor deposition method, the vaporization source is vaporized by the electron beam and vapor deposition is carried out. Accordingly, the purity of the film formed is dependent upon the purity of the target plate or vaporized source. Thus, to obtain a high purity of the metallic tantalum or Ta_2O_5 films, it is essential for the sputter target plate or electron beam vaporization source to have a high purity. In this specification, the tantalum target includes tantalum materials of all shapes used for the sputtering source or vaporization source.

The tantalum targets that are commercially available now are prepared by molding, sintering and dissolution of commercially available metallic tantalum powder with a purity of 99.9%, followed by machining. Impurity contents are usually below 100 ppb for alkali metals such as Na, K, etc., below 5 ppb for radioactive elements such as U, etc., and below 1 ppm for transition metals such as Fe, Ni, Cr, Mn, etc., which are quite low. However, further reduction in the impurities is desired with stable reliability.

Another important point is that, more recently, the presence of high-melting metallic impurities such as Nb, Mo, W, Zr, Hf, etc. has also attracted attention, due to the new realization that the oxides of high-melting metals, especially molybdenum and tungsten oxides, have a high electric conductivity and cause the leakage current of tantalum films.

The tantalum targets that are available commercially have an Nb content of several tens of ppm, and 1-10 ppm of Mo, W, Zr, etc. Such a content of high-melting metallic impurities is more than two-fold higher compared with other LSI constitutional materials such as silicon, molybdenum, etc. Thus, unless such content is lowered by more than two-fold, they are basically not suitable as the electronic device element constitutional materials requiring high reliability.

Presentation of the invention

Under such circumstances, an objective of the present invention is to develop a high-purity metallic tantalum target with a low content of not only alkali metals, radioactive metals and transition metals, but also all high-melting metals, and also to establish a method for its manufacture.

As a result of an investigation for achieving such objectives, we have reached a conclusion that, to remove the high-melting metallic impurities, it is essential that wet purification process be used for removing high-melting metallic impurities from the tantalum

(compound) powder that is available commercially. While alkali metals and transition metals can be removed by the electron beam melting process, high-melting metals cannot be removed by electron beam melting. Several methods can be considered for wet purification, e.g., ① the ion exchange method and ② solvent extraction method. In the ion exchange method ①, separation of Nb, Mo, W, Zr, and Hf is possible, but since the adsorption target element is Ta, the cost is high and productivity is considered to be low. In the solvent extraction method ②, using MIBK as the extractant is known industrially, but it has a low flash point, thus safety is a concern.

Considering that the most suitable wet process is employing the fractional crystallization process involving preferential crystallization of a tantalum compound by utilizing the solubility difference, followed by reduction of the thus obtained tantalum compound to obtain tantalum powder, we carried out a further study. As a result, a very high purification effect is obtained by crystallization of tantalum potassium fluoride (K_2TaF_7) and sodium reduction.

Through the molding-sintering-melting ingot making process and machining, final tantalum targets are obtained from the high-purity tantalum powder thus obtained by such a wet purification process. Higher purity can be obtained by using the electron beam process for melting.

Finally, ultra-high-purity tantalum target can be obtained with the content of harmful impurities reduced to a trace level.

Thus, the present invention concerns a high-purity metallic tantalum target and a process for its production, characterized by the alkali metal content being below 50 ppb, the radioactive element content being below 5 ppb, the transition metal content being below 3 ppm and the high-melting metal content being below 3 ppm, wherein (A) metallic tantalum or tantalum pentoxide is dissolved in hydrofluoric acid or a mixed acid containing hydrofluoric acid to obtain a tantalum-containing aqueous solution; (B) the tantalum-containing aqueous solution is treated with an aqueous solution containing potassium ions to precipitate tantalum potassium fluoride; (C) the tantalum potassium fluoride crystals are collected and reduced by sodium to obtain a product containing metallic tantalum powder, potassium fluoride and sodium fluoride; (D) the product is washed to recover the metallic tantalum; (E) the recovered metallic tantalum powder is press-molded, sintered, then melted to obtain a metallic tantalum ingot; (F) the metallic tantalum ingot is machined to obtain targets. Between processes B and C, if needed for further improvement in the purification effects, a process may be added involving addition of the tantalum potassium fluoride crystals to an ammonia solution to obtain tantalum hydrate, dissolving the separated tantalum hydrate in hydrofluoric acid to obtain a tantalum-containing aqueous solution, and treating the tantalum aqueous solution with an aqueous solution containing potassium ions to precipitate tantalum potassium fluoride crystals.

In the present invention, the alkali metals indicate the metals belonging to Periodic Group IA, such as Na, K, and Li. The radioactive elements indicate elements having radioactivity such as U, Th, etc. The transition metals indicate those elements having relatively low melting points broadly among the transition metals and including Fe, Cr, Ni, and Mn. The high-melting metals indicate those elements having relatively high melting points, broadly among the transition metals including Nb, Mo, W, Zr and Hf.

Brief description of figures

Figure 1 is a basic flow sheet of the method for manufacturing the tantalum target of the present invention.

Figure 2 is a flow sheet of the application example covering from the raw material Ta_2O_5 to K_2TaF_7 crystal preparation.

Figure 3 is a flow sheet of the application example covering from the K_2TaF_7 crystals to the Ta powder preparation.

Optimal embodiment of the invention

Figures 1-3 illustrate basic flow sheets and preferred methods for practicing the present invention. The following explanation is based on these figures.

As described above, the tantalum targets that are commercially available contain up to several tens of ppm of high-melting metal impurities. The purification of such high-melting metal impurities involves wet purification process, starting with dissolution of the starting materials such as metallic tantalum or tantalum compounds such as tantalum pentoxide that are available commercially to obtain aqueous solutions. In summary (see Figure 1), the impurities that are removed in the wet purification process are mainly high-melting metals and radioactive elements, resulting in tantalum potassium fluoride (K_2TaF_7) crystals. Next, the crystals are reduced by sodium to obtain high-purity tantalum powder. The high-purity tantalum powder is then passed through drying, molding, sintering (preferably cold isostatic pressing (CIP)) and hot isostatic pressing (HIP) and melting (preferably electron beam (EB) melting). Here, the elements that have a higher vapor pressure than tantalum, such as alkali metals, radioactive elements and transition metals, are removed. The obtained tantalum ingot is machined to form a tantalum target.

The characteristics of the present invention basically involve the combination of a wet purification process and a dry treatment process. In the former, radioactive elements and high-melting metals are removed in particular, while in the latter, alkali metals, radioactive elements and transition metals are removed in particular.

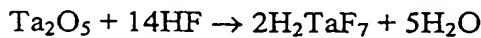
Each process is explained separately (see Figure 2 and 3)

(I) Tantalum raw material

The tantalum raw material that is readily soluble in acids or alkalis should have the highest purity possible. The raw materials are mainly metallic tantalum powder or tantalum pentoxide (Ta_2O_5), which are commercially available. Other tantalum compounds such as tantalum potassium fluoride, etc. can also be used.

(II) Dissolution

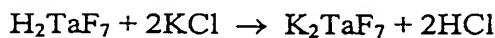
The dissolution may be performed in alkalis and various mineral acids, while using hydrofluoric acid, which is capable of dissolution at a relatively low temperature of below 100°C and producing K_2TaF_7 crystals, is preferred. Basically, metallic tantalum powder is dissolved in a mixture of hydrofluoric acid and nitric acid, etc., and Ta_2O_5 powder is dissolved in hydrofluoric acid. In the case of Ta_2O_5 , the following reaction occurs:



The amount of hydrofluoric acid used should be above the theoretical amount, more preferably 1.3 to 1.5 times the theoretical amount. The dissolution temperature should be above 60°C, preferably above 80°C. With a smaller amount of hydrofluoric acid or lower dissolution temperature, Ta_2O_5 dissolution is poor, thus the yield is affected.

(III) Crystal recovery

The tantalum-containing aqueous solution is filtered from the solid impurities, and the filtrate is gradually added to a solution containing potassium ions, such as from KCl , etc. The following reaction with K_2TaF_7 occurs:



The amount of potassium chloride used in the crystallization of tantalum potassium fluoride should be above 1 time, preferably 1.1-1.4 times the theoretical amount. The temperature should be above 60°C, preferably 80-95°C. With a smaller amount of potassium chloride, the crystallization yield of the tantalum potassium fluoride is small, while at a lower temperature, fine tantalum potassium fluoride crystals are formed, which results in poor filterability and washability.

After filtration, the tantalum potassium fluoride crystals are washed sufficiently with a potassium fluoride solution to a pH of about 5-6, then dried.

When further purification is needed, a process is carried out involving addition of the tantalum potassium fluoride crystals to an ammonia solution to obtain tantalum hydrate, dissolving the separated, washed and dried tantalum hydrate in hydrofluoric acid to obtain a tantalum-containing aqueous solution, and treating the tantalum aqueous solution with an aqueous solution containing potassium ions to precipitate tantalum potassium fluoride crystals. After washing and filtration, washing with a potassium fluoride solution is carried out, followed by drying. If needed, this process is repeated.

In the above wet process, the handling equipment such as the reaction container, filter, etc. should be clean and chemically inert. Water and chemicals of high purity are preferred.

(IV) Reduction

The tantalum potassium fluoride crystals that are obtained in the above wet process are reduced. The tantalum potassium fluoride is easily reduced by highly active metals such as Ti, Al, Mg, Na, etc., while Na is generally used as a reducing agent in industrial operation. In the present invention, the Na reduction method is employed taking into account that Na does not form alloys with Ta and does not contaminate the product metallic Ta, and the by-product NaF can be removed relatively easily.

A conventional Na reduction method can be used without any special restrictions. The reaction between tantalum potassium fluoride and Na is shown below, and it is an exothermic reaction:



In the above reaction, to stabilize the reaction by lowering the reaction temperature, usually, alkali halides such as NaCl, etc. are added as diluents.

After the reaction, the reaction product containing metallic tantalum powder, sodium fluoride, and potassium fluoride deposit on the bottom of the reaction container.

The recovered reaction product is first washed with ethanol by the usual manner to remove unreacted metallic sodium as sodium ethylate, then sodium fluoride and potassium fluoride are removed by hot water. After obtaining the metallic tantalum powder, appropriate washing is carried out such as aqua regia washing, fluoric acid washing, ammonia washing, acetone washing, etc., followed by drying.

The cleanliness of the reaction containers, chemicals and environment is similar as in the case above.

(V) Press molding, sintering and melting

To prepare the tantalum ingot, the high-purity metallic tantalum powder that is obtained by reduction of tantalum potassium fluoride is subjected to an appropriate ingot formation treatment. During the melting, it is necessary to remove the alkali metals, radioactive elements and transition metals. Here, while purification technology such as vacuum melting, etc. suitable for removing impurities can be used, the electron beam melting method with a high degree of impurity removal is preferred.

The molding used in the electron beam melting should be free from interior trapped gases and should not have the danger of contamination or have a high bulk density.

For example, tantalum powder is packed in a cylinder made of Teflon and subjected to cold isostatic pressing under a pressure of 1000-1600 kg/cm². The molding thus obtained is placed in a mild steel container, heated at high temperature, and the mild steel container is sealed

under vacuum degassing. The sealed mild steel container is placed in a hot isostatic pressing apparatus and sintered under the conditions of a temperature of 1000-1500°C and pressure of 900-1500 kg/cm². After cooling, the tantalum sinter is taken out of the mild steel container. The sinter is an electrode suitable for electron beam melting. Next, the tantalum sinter electrode is subjected to electron beam melting to form a tantalum ingot. If needed, the electron beam melting is repeated 2-3 times.

(VI) Machining

Finally, the obtained tantalum ingot is machined to form a tantalum target in the desired shape. Plastic working, cutting, and surface finishing can be done by conventional methods with careful contamination prevention.

The ultra-high-purity metallic tantalum target thus obtained has an alkali metal content below 50 ppb (0.05 ppm), radioactive element content below 5 ppb (0.005 ppm), transition metal content below 3 ppm, and high-melting metal content below 3 ppm.

More specifically, high-purity tantalum targets with the following impurity contents can be obtained in stable manner:

Alkali metals	Na	<0.02 ppm
	K	<0.02 ppm
Radioactive elements	U	<0.001 ppm
Transition metals	Fe	<0.05 ppm
	Ni	<0.02 ppm
	Cr	<0.02 ppm
High-melting metals	Nb	<0.2 ppm
	Mo	<0.2 ppm
	W	<0.3 ppm
	Zr	<0.2 ppm

The effects of the present invention are summarized below.

By using this tantalum target, for example, Ta₂O₅ films can be obtained by sputtering in an argon-oxygen gas mixture. Since the Ta₂O₅ film does not contain any harmful impurities, high performance and operation reliability of the semiconductor devices can be assured. The MOS capacitance of a Al/Ta₂O₅/p-Si structure, prepared by reaction sputtering in Ar-O₂ mixed gas using the tantalum target of the present invention, showed a very small leakage current compared to that of conventional ones.

The present invention provides ultra-high-purity targets in which the effects of alkali metals, radioactive elements, transition metals and high-melting metals have been reduced to a negligible level in semiconductor devices and, through development of a reliable manufacturing method for such targets, makes a contribution to the progress of the electronics industry by enabling the preparation of highly reliable semiconductor device parts such as insulation films, electrodes, etc.

Application example

11 kg of commercially available tantalum pentoxide and 18 kg of high-purity 50% hydrofluoric acid were stirred in a Teflon reaction bath at 80°C for 10 h for dissolution, then the mixture was filtered through a 0.2 µm Teflon Millipore filter to separate the solid impurities and obtain a tantalum-containing aqueous solution.

Next, 3 kg of special grade potassium chloride were dissolved in 18 L of ultra-pure water, heated to 80°C, and treated gradually with 6 L of the tantalum-containing aqueous solution to precipitate the tantalum potassium fluoride. The mixture was filtered through a Teflon cloth, and the tantalum potassium fluoride crystals were washed with a 100 g/L potassium fluoride aqueous solution.

Next, the tantalum potassium fluoride crystals were dried and reduced by metallic sodium in an iron reaction container under heating to form metallic tantalum powder, potassium fluoride and sodium fluoride. Namely, 5.50 kg of tantalum potassium fluoride and 2.0 kg of metallic sodium were heated together under an argon gas atmosphere to 800°C then held at 800°C for 3 h for reduction, followed by removing excess Na by alcohol, removing potassium fluoride and sodium fluoride by washing with hot water, washing with aqua regia and hydrofluoric acid for removal of impurities, and drying to obtain metallic tantalum powder.

This metallic tantalum powder was sintered by cold isostatic pressing (1500 kg/cm²) and hot isostatic pressing (1000 kg/cm², 1400°C, 1 h), melted by electron beam, and machined to obtain a high-purity metallic tantalum target. The impurities content of this high-purity metallic tantalum target is given in Table I.

Table I (unit: weight ppm)

	Na/Ta	K/Ta	U/Ta	Fe/Ta	Ni/Ta	Cr/Ta	Nb/Ta	Mo/Ta	W/Ta	Zr/Ta	Si/Ta
① 原料五酸化タントタル	—	—	<0.002	<24	0.05	0.1	1.0	0.24	2.9	<0.6	—
② フッ化タントタルカリウム精晶	—	—	<0.003	<3	—	—	<1	<0.4	<0.6	<0.4	—
③ 金属タントタル粉末	60	52	0.001	110	0.7	0.8	<0.2	<0.2	<0.3	<0.2	—
④ 本格明高純度金属タントタル製ターフェット	<0.02	<0.02	<0.01	<0.02	<0.02	<0.02	<0.2	<0.2	<0.3	<0.2	0.1
⑤ 市販金属タントタル製ターフェット	<0.05	<0.05	<0.001	0.1	—	—	5.0	2	10	1	—

Key: 1 Tantalum pentoxide raw material
 2 Tantalum potassium fluoride crystal
 3 Metallic tantalum powder
 4 High-purity metallic tantalum target of the present invention
 5 Commercially available metallic tantalum target

As clearly shown in Table I, the purity of the high-purity metallic tantalum targets obtained in the present invention is above 99.999%. Compared with commercially available tantalum targets, the high-purity metallic tantalum targets obtained had contents of high-melting metals such as Nb, Mo, W, Zr, etc. of below 1 ppm each. No Si was present.

Industrial application possibilities

By using the ultra-high-purity tantalum targets of the present invention, impurities harmful to semiconductor device operation are removed to a negligible level, and as a result, high-quality, high-reliability LSI Ta_2O_5 dielectric films and metallic Ta electrode films such as LSI gate electrodes, source electrodes, drain electrodes, etc. can be formed, and the reliability of operation of the semiconductor devices with ever-increasing integration and density can be enhanced.

Claims

1. High-purity metallic tantalum target, characterized by having alkali metal content below 50 ppb (0.05 ppm), radioactive element content below 5 ppb (0.005 ppm), transition metal content below 3 ppm and high-melting metal content below 3 ppm.
2. High-purity metallic tantalum target of Claim 1, wherein the alkali metal content is below 20 ppb (0.02 ppm), the radioactive element content is below 1 ppb (0.001 ppm), the transition metal content is below 0.1 ppm and the high-melting metal content is below 0.3 ppm.
3. Method for the manufacture of high-purity metallic tantalum target with an alkali metal content below 50 ppb, radioactive element content below 5 ppb, transition metal content below 3 ppm and high-melting metal content below 3 ppm, wherein (A) metallic tantalum or tantalum pentoxide is dissolved in hydrofluoric acid or a mixed acid containing hydrofluoric acid to obtain a tantalum-containing aqueous solution; (B) the tantalum-containing aqueous solution is treated with an aqueous solution containing potassium ions to precipitate tantalum potassium fluoride; (C) the tantalum potassium fluoride crystals are collected and reduced by sodium to obtain a product containing metallic tantalum powder, potassium fluoride and sodium fluoride; (D) the product is washed to recover the metallic tantalum; (E) the recovered metallic tantalum powder is press-molded, sintered, then melted to obtain a metallic tantalum ingot; (F) the metallic tantalum ingot is machined to obtain a high-purity metallic tantalum target.

4. Method according to Claim 3, wherein the melting following the press molding and sintering is done by cold isostatic pressing, hot isostatic pressing and electron beam melting.

5. Method for the manufacture of high-purity metallic tantalum target with an alkali metal content below 50 ppb, radioactive element content below 5 ppb, transition metal content below 3 ppm and high-melting metal content below 3 ppm, wherein (A) metallic tantalum or tantalum pentoxide is dissolved in hydrofluoric acid or a mixed acid containing hydrofluoric acid to obtain a tantalum-containing aqueous solution; (B) the tantalum-containing aqueous solution is treated with an aqueous solution containing potassium ions to precipitate tantalum potassium fluoride; (C) the tantalum potassium fluoride crystals are added to an ammonia solution to obtain tantalum hydrate, the separated tantalum hydrate is dissolved in hydrofluoric acid to obtain a tantalum-containing aqueous solution, and the tantalum aqueous solution is treated with an aqueous solution containing potassium ions to precipitate tantalum potassium fluoride crystals, this process being able to be repeated as needed; (D) the tantalum potassium fluoride crystals are collected and reduced with sodium to obtain a product containing metallic tantalum powder, potassium fluoride and sodium fluoride; (E) the product is washed to recover the metallic tantalum; (F) the recovered metallic tantalum powder is press-molded, sintered, then melted to obtain a metallic tantalum ingot; (G) the metallic tantalum ingot is machined to obtain a high-purity metallic tantalum target.

6. Method according to Claim 5, wherein the melting following the press molding and sintering is done by cold isostatic pressing, hot isostatic pressing and electron beam melting.

7. High-purity metallic tantalum target prepared by the method of Claim 3 or 5.

8. Method for forming high-purity Ta_2O_5 film by reaction sputtering of the target of Claim 1 in an inert gas-oxygen mixed gas.

9. Method for forming high-purity metallic tantalum film by sputtering using the target of Claim 1.

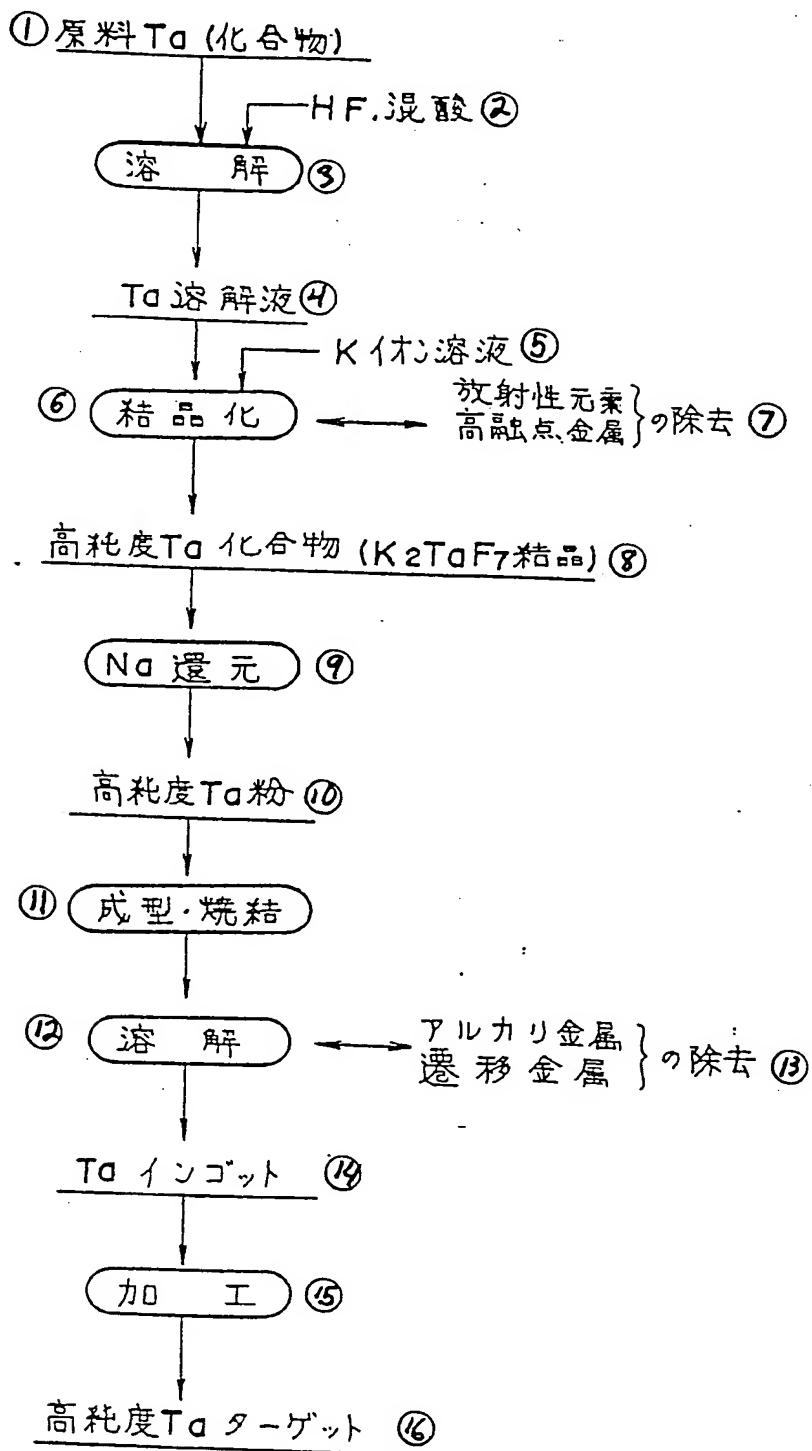


Figure 1

Key: 1 Raw material Ta (compound)
2 Mixed acid
3 Dissolution
4 Solution
5 Ion solution
6 Crystallization
7 Removal of radioactive elements and high-melting metals
8 High-purity Ta compound (K_2TaF_7 crystal)
9 Na reduction
10 High-purity Ta powder
11 Molding, sintering
12 Melting
13 Removal of alkali metals and transition metals
14 Ta ingot
15 Machining
16 High-purity Ta target

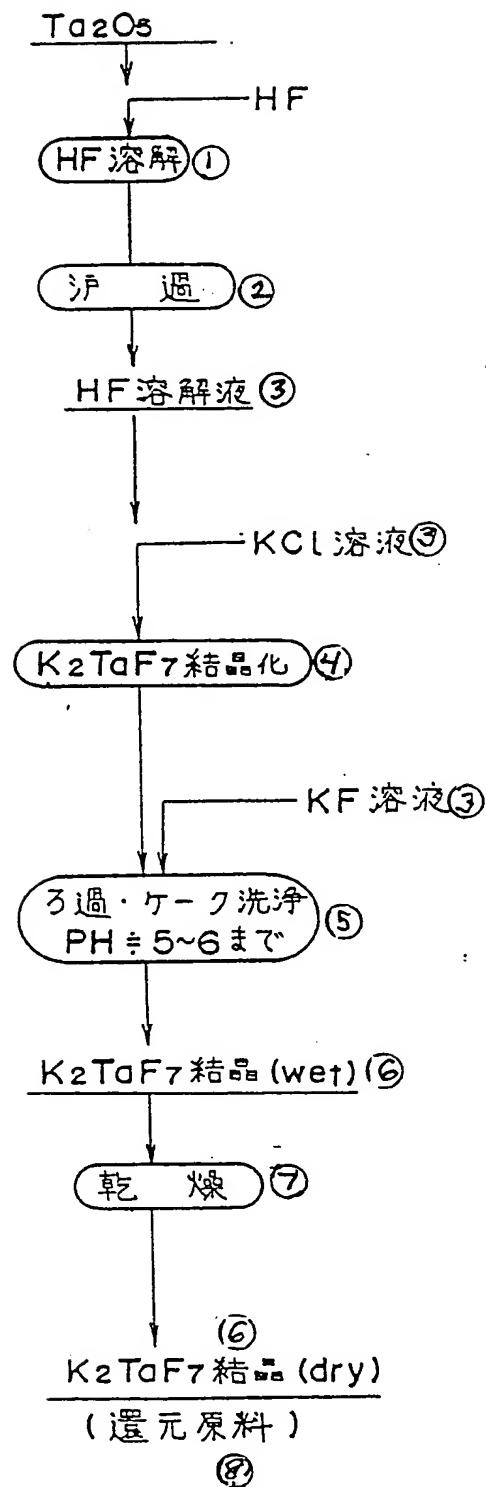


Figure 2

Key: 1 Dissolution
2 Filtration
3 Solution
4 Crystallization
5 Filtration, cake washing to pH of about 5-6
6 Crystals
7 Drying
8 Reduction starting material

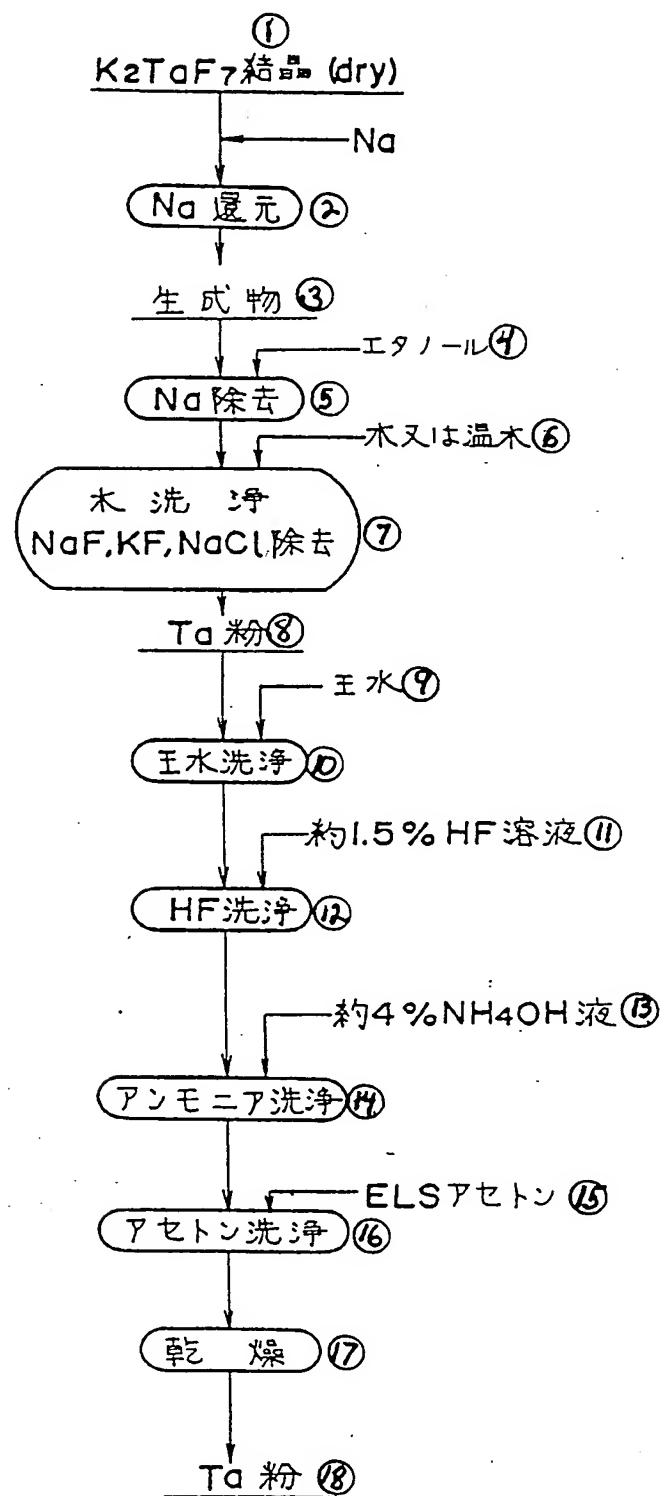


Figure 3

Key:	1	Crystal
	2	Reduction
	3	Product
	4	Ethanol
	5	Removal
	6	Water or hot water
	7	Removal by water washing
	8	Powder
	9	Aqua regia
	10	Aqua regia washing
	11	About 1.5% HF solution
	12	Washing
	13	About 4% NH ₄ OH solution
	14	Ammonia washing
	15	Acetone
	16	Acetone washing
	17	Drying

